



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE PCT NATIONAL STAGE APPLICATION OF
PETR KVITA ET AL

INTERNATIONAL APPLICATION NO. PCT/EP 02/14785

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FOR: PARTICULATE COMPOSITION
COMPRISING DYE FIXATIVES
U.S. APPLICATION NO: 10/500,778

35 USC 371 DATE: JULY 1, 2004

Group Art Unit 1796
Examiner: A. S. Khan

DECLARATION UNDER RULE 132

I, HAUKE ROHWER, a citizen of Germany, residing at 44, Talweg, 79540 Loerrach, Germany, hereby declare:

That I was awarded the degree of a Dipl. Laboratory Chemist, University Siegen, Germany, in 1989;

That I have been employed by Ciba as a chemist since 1989 and presently hold the position of an Application Laboratory Head in the Segment Plastic Additives;

That I have been engaged in the field of Home & Fabric Care for Ciba since 1997;

That based on the above education and experience, I consider myself an expert in the field of Fabric Care (detergents and rinse conditioners).

I, HAUKE ROHWER, declare that the preparation of the compositions (A), (B) and (C) as indicated in Part I of this Declaration as well as the following determination of dye transfer inhibition indicated in Part II of this Declaration were carried out under my direction and supervision;

That I am submitting herewith the following exact report of the preparations and tests mentioned below.

I, HAUKE ROHWER, declare that the following tests were carried out under my direction and supervision. That I am submitting herewith the following exact report of the tests and the results obtained.

Part I
Dye fixative material

Composition (A) was prepared according to the prescription given in Example 9 on pages 34 and 35 of the instant invention.

Composition (B) was prepared according to the prescription given in U.S. Patent No. 6,187,740, column 30, lines 1 to 18, containing as PVP (polyvinylpyrrolidone) the commercial product K-30 ex ISP Chemical Products as fixing agent or dye transfer inhibiting agent.

Composition (C) was prepared according to the prescription given in U.S. Patent No. 6,187,740, column 28, lines 6 and 7, containing a co-polymer of PVP (polyvinylpyrrolidone) and PVI (polyvinylimidazol) which corresponds with the commercial product SOKALAN HP56 from BASF, Germany, as dye fixing agent or dye transfer inhibiting agent.

All compositions were prepared as granules as given in Example 9 of the instant invention in order to have the comparison at the point of closest approach: Composition (A) is free from PVP, whereas the composition (B) contains PVP and the composition (C) contains a co-polymer of PVP (polyvinylpyrrolidone) and PVI (polyvinylimidazole), respectively. The compositions (A), (B) or (C), respectively, are mixed with a standard detergent in such a way that the amount of the active substance is:

Composition	Granule composition mixed with standard detergent containing
(A)	1% TINOFIX® CL
(B)	1% K-30 (PVP)
(C)	1% SOKALAN HP56 (PVP/PVI)

The standard detergent consists of:

	ECE 77 - ISO 105-C06
Sodiumalkylbenzenesulfonate	8.0
Alcoholethoxylate C ₁₄ -C ₁₅ EO 7-8	2.9
Soap	3.5
Magnesiumsilikate	1.9
Sodiumtriposphosphate	43.8
Sodiumsilikate	7.5
EDTA	0.2
CMC	1.2
Sodiumsulfate	21.2
Water	9.8

The resultant detergent compositions (A), (B) and (C) contain 1 weight % of the active substance TINOFIX CL® (composition A), 1 weight % of the active substance PVP of K-30 (composition B) and 1 weight % of the co-polymer of the active substances PVP and PVI based on the commercial product SOKALAN HP56 (composition C) as given above.

I, HAUKE ROHWER, declare that the above detergent compositions (A), (B) and (C) exhibited the same relative purity.

Part II

I) Determination of dye fixation and dye transfer inhibition

The detergent compositions were used for the preparation of the following washing compositions: Tap water and 2.5 g/l of each of the above mentioned detergent composition (A), (B) or (C), respectively, containing detergent and dye fixative agent in composition (A), detergent and PVP in compositions (B), or detergent and PVP/PVI in composition (C), respectively, were used. 5 g of a cotton fabric dyed with 2.09% SOLOPHENYL® Blue 4GL (bleeder fabric) were washed together with 5 g of bleached white cotton fabric (accompanying fabric). The liquor ratio was 1:10 (tap water), the washing temperature was 40°C and the duration of washing in an ATLAS Linitest (ATLAS; Chicago, Illinois) was 15 minutes. The fabrics were then rinsed for 30 seconds under running tap water, spun and dried.

The extinction of each wash bath was determined with an Uvikon 810 Photometer and the Delta E of each accompanying fabric compared with the starting material was determined as given in the instant invention on pages 37 to 39 of the English text (light absorption of the liquor according to the Beer-Lambert law which teaches that extinction at constant wavelength and pathlength is a direct measure for the concentration of a product in solution. Extinction = $E \cdot c \cdot l$, with E being the extinction coefficient, c being the concentration, l being the pathlength of the light through the solution).

Delta E Measurement

The reflectance spectra of the accompanying fabrics were measured prior to and after the wash. The spectra were transformed into the corresponding L, a, b values according to the CIE standard procedure [1]. Using the L, a, b values of a swatch before (L_0 , a_0 , b_0) and after the wash (L_1 , a_1 , b_1) the ΔE value was calculated using the following formula:

$$\Delta E = [(a_0 - a_1)^2 + (b_0 - b_1)^2 + (L_0 - L_1)^2]^{1/2}$$

- [1] Supplement No.2 of CIE Publ. No.15 (E-1.3.1) (1971) Bureau Central de la CIE, Paris 1978a

II) Evaluation

The extinction of the wash liquor was determined at 600 nm (Uvikon 810) after the wash. The Delta E of each accompanying fabric compared with the starting material was determined by a Spectraflash 500 spectrophotometer.

A high value of the extinction means a low value of degree of fixation effect on the fabric and a high concentration of the dye in the wash liquor; and a high Delta E value means a low degree of dye transfer inhibition effect and a high amount of dye fixed on the accompanying fabric.

Results

Table 1

	Extinction	Delta E
Detergent Composition (A)	0.86	32
Detergent Composition (B)	1.15	36
Detergent Composition (C)	1.45	31

As evident from the Table 1, the composition (A), supra, is superior to the closest composition containing PVP (polyvinylpyrrolidone) or the composition containing PVP/PVI

(co-polymer of polyvinylpyrrolidone and polyvinylimidazole) of the prior art as given in Part I with regard to the measured extinction value. Composition (A) exhibits the clearly lower extinction value and that means the wash liquor exhibits the smallest amount of SOLOPHENYL® Blue 4GL in the wash liquor.

The Delta E values exhibit the amount of SOLOPHENYL® Blue 4GL transferred through the wash liquor to the accompanying fabric and fixed onto this fabric. The amount of SOLOPHENYL® Blue 4GL transferred through the wash liquor to the accompanying fabric and fixed onto this fabric is very similar in compositions (A) and (C), whereas the amount of SOLOPHENYL® Blue 4GL transferred through the wash liquor to the accompanying fabric and fixed onto this fabric in composition (B) means a lower degree of dye transfer inhibition effect and a higher amount of dye fixed on the accompanying fabric.

The value of the extinction of composition (A) is distinct superior to the value of the extinction of the compositions (B) and (C) of the prior art. Thus, the known compositions (B) and (C) containing as active substance PVP (polyvinylpyrrolidone) or the co-polymer of PVP (polyvinylpyrrolidone) and PVI (polyvinylimidazole) do not reach the effect obtainable with dye fixative material TINOFIX® CL as given in composition (A).

The comparison of the Delta E values of compositions (A), (B) and (C) show a similar or higher value which means a similar or lower degree of dye transfer inhibiting effect of SOLOPHENYL Blue 4GL transferred onto on the accompanying fabric.

As a consequence from the test results obtained, the composition containing TINOFIX® CL clearly shows a better fixation of the dye on the original dyed fabric which fact is demonstrated with a lower extinction value and which means that the bleeding of the dyed fabric was inhibited in a clearly better way than demonstrated with the compositions (B) and (C). Compositions (B) and (C) despite the high amount of dyestuff SOLOPHENYL Blue 4GL within the washing liquor (extinction value) demonstrate a similar or a little better dye transfer inhibition from the dyed fabric to the undyed fiber material (Delta E value). However, in washing compositions (B) and (C) a high amount of dyestuff SOLOPHENYL Blue 4GL is retained in the liquor.

Frequently washing with a detergent and TINOFIX® CL will clearly result in a more coloured dyed fabric compared with washing result with a detergent and PVP or PVP/PVI because bleeding of the dyed fabric treated with compositions containing PVP or PVP/PVI is much stronger than bleeding of the dyed fabric treated with a composition containing TINOFIX® CL.

Part III

I, HAUKE ROHWER, hereby declare:

1. That based on my education and experience, I consider myself an expert in the field of Fabric Care;
2. That the results of the above tests show that the composition (A) according to the instant invention is superior to the closest compositions (B) or (C) of the prior art with respect to the properties tested (dye fixation on dyed fabric material);
3. That dye fixation on dyed fabric material is an important feature for the industry and an improvement in this property is of considerable importance;
4. That the above given measurement of the dye fixation demonstrates a significant improvement in this property which is of commercial importance;

5. That the improvement attainable with respect to dye fixation on dyed fabric material could not be foreseen and the results of the tests are surprising to me and I would never have predicted such difference in the property tested.

I, HAUKE ROHWER, declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 10th day of September 2008



HAUKE ROHWER